Science of the Total Environment 957 (2024) 177483

Contents lists available at ScienceDirect

Science of the Total Environment

journal homepage: www.elsevier.com/locate/scitotenv

Optimising microplastic polyethylene terephthalate fibre extraction from sediments: Tailoring a density-separation procedure for enhanced recovery and reliability

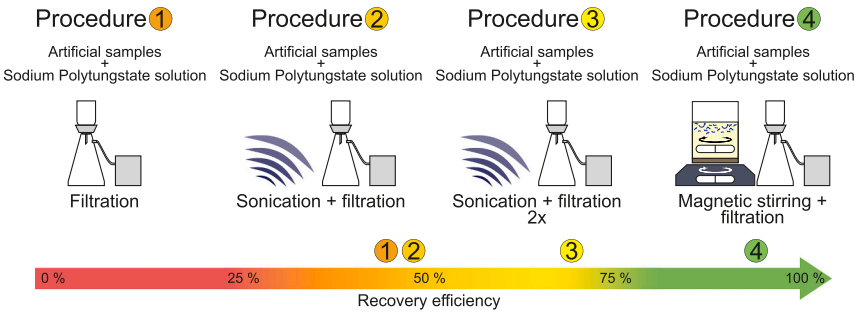
Francesca Uguagliati [a,*], Massimiliano Zattin [a], Kryss Waldschläger [b], Massimiliano Ghinassi [a]

[a] University of Padova, Department of Geosciences, Via Gradenigo 6, 35131 Padova, Italy
[b] Wageningen University and Research, Hydrology and Environmental Hydraulics Group, Wageningen, the Netherlands

HIGHLIGHTS

- The lack of standardised methods for microplastic extraction hinders result comparability.
- Sodium Polytungstate solution effectively extracts PET fibres from sediments.
- PET fibres can be recovered from sandy, mud-free sediments with a simple procedure.
- Microplastic extraction from mud-rich sediments requires proper measures.

GRAPHICAL ABSTRACT



ARTICLE INFO

Editor: Damià Barceló

Keywords:
Microplastics
Extraction protocol
Density separation
Sodium polytungstate

ABSTRACT

Despite the presence of microplastics in sediments being widely acknowledged, the absence of standardised processing methods in extracting microplastics can compromise reliable and comparable results. Density separation is a predominant method for extracting microplastics from sediments. In this study, Sodium Polytungstate (ρ = 1.6 g cm^{-3}) was selected as the density separation agent for three key factors: i) optimal density for extracting common plastic polymers, ii) low toxicity, and iii) recycling potential of the solution. It is therefore cost-effective, and the risk of solution dispersal is minimal. The solution was tested through four separation procedures, extracting PET fibres from three artificial sediment mixtures (i.e., pure sand, pure mud, and 50 % sand and 50 % mud). The results indicate that the solution employed in this work is highly effective for extracting microplastic fibres from sediments, with recovery rates up to 99 %. However, the results highlight differences in the recovery among the four procedures and in terms of the sediment characteristics. Specifically, extracting microplastics was easier in sandy sediment samples than in mud-rich ones. The complexity of extracting microplastics from mud-rich sediments results from i) the creation of microplastic-sediment aggregates forming denser structures, that settle down trapping microplastics in sediments; ii) the development of a clay sediment cap that hinders the rise of microplastics to the surface. Reducing the risk of underestimation of microplastic content in mud-rich samples can be accomplished by applying a procedure that involves placing the samples with the Sodium Polytungstate solution on a stirring plate while progressively lowering the rotation velocity. Using this method, cohesive sediments lose their ability to trap microplastics while aggregating,

* Corresponding author.
E-mail address: francesca.uguagliati@phd.unipd.it (F. Uguagliati).

https://doi.org/10.1016/j.scitotenv.2024.177483
Received 1 July 2024; Received in revised form 1 November 2024; Accepted 8 November 2024
Available online 16 November 2024

consequently reducing their ability to drag microplastics to the bottom. This facilitated microplastics to reach the liquid surface, thereby enabling an efficient retrieval even in mud-rich samples.

1. Introduction

The production of plastic has consistently increased since the 1950s, and a large amount of plastic introduced into the environment may end up in the oceans (Lebreton and Andrady, 2019). The growth of plastic production is causing significant concerns, as its impacts on ecosystems and organisms are acquiring increasing awareness, not just within the scientific community but also among the media and politics (Kumar et al., 2021). When exposed to environmental conditions, plastic waste undergoes weathering, breaking down into smaller pieces known as microplastics (MPs), ranging between 1 μm and 5 mm (Barnes et al., 2009, O'Brine and Thompson, 2010; Browne et al., 2011). MPs present a significant concern as they can adsorb and transport heavy metals and other pollutants and, when further disaggregated, release them into the environment (Brennecke et al., 2016; Jiang, 2018; Godoy et al., 2019; Liu et al., 2021). Additionally, the additives incorporated into plastics during manufacturing, such as plasticisers, flame retardants, colourants, and stabilisers, aimed at improving their properties, may be leached out during degradation, presenting a potential hazard (Bridson et al., 2023; Yu et al., 2024). Moreover, the small size of these particles makes them easily ingestible by organisms, further threatening ecosystems, biodiversity, and local economies (Wright et al., 2013; Kumar Chaudry and Sachdeva, 2021; Ghosh et al., 2023). Given their high mobility and long residence times (Padervand et al., 2020; He et al., 2021; Han et al., 2022), MPs are ubiquitous and have been labelled as emerging contaminants (Cole et al., 2011; Hamid et al., 2018; Akanyange et al., 2022).

Once introduced into the aquatic environment, sediments are a major sink of MP particles (Van Cauwenberghe et al., 2015; Yao et al., 2019; Darabi et al., 2021; Yang et al., 2021). To comprehend their patterns of accumulation over time, evaluate their toxic effects and develop mass balance models based on their fate and fluxes, reliable monitoring of their concentrations needs to be achieved (Kooi et al., 2016; De Arbeloa and Marzadri, 2024). However, standardised extraction and analysis methodologies to conduct a comprehensive assessment of these particles in sedimentary environments are still lacking (Bellasi et al., 2020; Bellasi et al., 2021). In the frame of extracting MPs from sediment samples, new techniques are constantly being developed due to the rapid growth in research in this field (Cutroneo et al., 2021). The absence of uniformity in processing environmental samples may result in relevant biases while comparing different study cases with similar features.

The current study examines extraction procedures, focusing on density separation, which is the most employed methodology among various techniques (Hanvey et al., 2017; Yao et al., 2019). The effectiveness of this method relies on the difference in density between sediments and MPs. Sediment density typically ranges around 2.5 to 2.7 g cm^{-3} (i.e., quartz density, which is by far the most abundant mineral in sands). In contrast, the density of common plastic polymers found in natural environments ranges from 0.2 g cm^{-3}, such as Expanded Polystyrene (EPS), to 1.4 g · cm^{-3}, including Polyethylene Terephthalate (PET) and Polyvinyl Chloride (PVC). Density separation works by mixing the sample with a high-density concentrated saline solution. After mixing, lighter particles tend to float to the top layer of the mixture, while denser ones sink to the bottom (Frias et al., 2018). Different saline solutions are employed in laboratories dedicated to MP research. However, numerous of these solutions exhibit significant disadvantages such as low density, elevated toxicity, and high costs (Quinn et al., 2017; Cutroneo et al., 2021; Kumar and Sharma, 2021; Zhang et al., 2021; Tirkey and Upadhyay, 2021; Ang et al., 2022; Mattsson et al., 2022; Schütze et al., 2022; Debraj and Lavanya, 2023). Since the density of the most common polymers does not overcome 1.4 g cm^{-3}, the density of the brine solution should exceed this value (Zhang et al., 2020). Additionally, the selected saline solution should possess low toxicity, be economically viable, and have the potential to be recycled and reused as this can significantly reduce the costs (Rodrigues et al., 2020). Table 1 presents the commonly used saline solutions, delineating their strengths and weaknesses. NaCl, NaI, and $ZnCl_2$ stand among the most extensively utilised salts despite exhibiting notable drawbacks, such as the relatively low density of NaCl (1.2 g cm^{-3}) and the high costs and toxicity associated with NaI and $ZnCl_2$ (Frias et al., 2018; Cutroneo et al., 2021; Tirkey and Upadhyay, 2021).

This study aims to assess the efficacy of Sodium Polytungstate (SPT hereinafter) in extracting MPs from clastic sand and mud. The selection of SPT was based on its ability to obtain an ideal density for the recovery of high-density common plastic polymers such as PET and PVC. SPT salt possesses the capacity to saturate in deionised water reaching a maximum density of 3.1 g cm^{-3} (Helbig and Pitt, 2018). Additionally, despite being expensive (Frias et al., 2018; Thomas et al., 2020), SPT exhibits the capability of being recovered and reused almost endless times by a process involving its filtration and water evaporation if diluted (Gregory and Johnston, 1987; Torresan, 1987; Krukowski, 1988). Another noticeable consideration is the low toxicity of SPT (Gregory and Johnston, 1987). While it poses harm to aquatic life with enduring effects (Eskola et al., 2021), the probability of environmental dispersion is significantly reduced due to its 100 % recyclability. It is also worth noting that SPT, unlike other high-density solutions, is non-irritant to the skin and does not form volatile vapours (Eskola et al., 2021). Considering the limitations of other salt solutions used in literature for density separation and the practical advantages offered by SPT, this study aims to test its recovery efficiency by using artificial sediment mixtures containing a controlled number of MP. Specifically, we test here the SPT capability to separate PET fibres from sediments ranging in grain size from mud (smaller than 63 μm) to medium sand (between 63 and 250 μm), which are widespread in natural and human-modified alluvial and coastal environments. The decision to employ PET fibres exclusively in our experiments is based on their dominance as the most common shape category of plastic polymer found in environmental samples (Burns and Boxall, 2018; Kooi and Koelmans, 2019). Additionally, PET is not only one of the most widespread plastic polymers but also possesses one of the highest densities among commonly encountered MPs (Rodrigues et al., 2019; Gazal and Gheewala, 2020). By demonstrating the effectiveness of our extraction method for PET fibres under these conditions, we ensure that it should also be capable of recovering fibres deriving from lower-density polymers. Additionally, modifications to the basic separation technique are suggested to optimise MP recovery rates in mud-rich sediments, emphasising the critical role of sediment grain size. This aspect is often overlooked in existing research, where MPs are frequently trapped within sediment aggregates (Besseling et al., 2017; Li et al., 2019; Waldschläger et al., 2022), leading to an overall underestimation of MPs content.

2. Materials and methods

2.1. Plastic-free sediments

Samples used in our experiments were obtained by adding MPs to plastic-free sediments. Plastic-free sediments were recovered in a quarry extracting sand and mud from fluvial Pleistocene deposits of the Upper Valdarno Basin (unit VRCd in Ghinassi et al., 2013). Sediments were collected at the quarry frontm below the modern soil) using a metal shovel after removing the outermost layer to prevent contamination.

Table. 1
Strengths and weaknesses of the most used saline solution for extracting microplastic particles from sediments.

Salt	Density ($g \cdot cm^{-3}$)	Strengths	Weaknesses	References
Calcium Chloride ($CaCl_2$)	1.3–1-5	Affordable. Not harmful to the environment and humans.	Low density to recover high-density polymers. Ca+ ions may react and aggregate organic matter.	Stolte et al., 2015; Crichton et al., 2017; Scheurer and Bigalke, 2018; Liu et al., 2019; Liu et al., 2020; Thomas et al., 2020; Zhang et al., 2020; Adams et al., 2021; Cutroneo et al., 2021; Kumar and Sharma, 2021; Schröder et al., 2021; Zhang et al., 2021; Ang et al., 2022; Duong et al., 2022; Razeghi et al., 2022; Debraj and Lavanya, 2023
Lithium metatungstate ($Li_6{\cdot}H_2W_{12}0_{40}$)	1.6–1.62	Ideal density to recover also high-density polymers.	Expensive. Moderate toxicity and hazard.	Chu et al., 2010; Frias et al., 2018; Masura et al., 2015; Quinn et al., 2017; Cutroneo et al., 2021; Eskola et al., 2021; Tirkey and Upadhyay, 2021; Razeghi et al., 2022
Monosodium Phosphate (NaH_2PO_4)	1.4–1.5	Affordable. Low toxicity.	Low density to recover high-density polymers. The need to heat the solution to increase the density during the processes complicates the extraction procedure.	Zhang et al., 2020; Cutroneo et al., 2021; Zhang et al., 2021; Bellasi et al., 2021; Ang et al., 2022; Debraj and Lavanya, 2023
Potassium Iodide (KI)	1.5–1.8	Ideal density to recover also high-density polymers.	High toxicity and hazard.	Mu et al., 2019; Scott and Green, 2020; Cutroneo et al., 2021; Eskola et al., 2021; Kurzweg et al., 2022; Santana et al., 2022
Sodium Bromide (NaBr)	1.37–1.5	Potential for solution recycling and reuse. Low toxicity.	Expensive.	Quinn et al., 2017; Frias et al., 2018; Liu et al., 2019; Liu et al., 2020; Thomas et al., 2020; Cutroneo et al., 2021; Halbach et al., 2021; Kumar and Sharma, 2021; Schütze et al., 2022
Sodium Chloride (NaCl)	1.15–1.3	Affordable and easily available. Low toxicity. Environmentally friendly.	Low density to recover high-density polymers. The need for multiple repetitive separation steps makes the process time-consuming.	Vianello et al., 2013; Masura et al., 2015; Van Cauwenberghe et al., 2015; Coppock et al., 2017; Sruthy and Ramasamy, 2017; Abidli et al., 2018; Frias et al., 2018; Pagter et al., 2018; Reed et al., 2018; Bayo et al., 2019; Blair et al., 2019; Filgueiras et al., 2019; Han et al., 2019; Liu et al., 2019; Stock et al., 2019; Ferreira et al., 2020; Liu et al., 2020; Mataji et al., 2020; Thomas et al., 2020; Wu et al., 2020; Zhang et al., 2020; Bellasi et al., 2021; Chouchene et al., 2021; Cutroneo et al., 2021; Nava and Leoni, 2021; Radford and Zapata-Restrepo, 2021; Tirkey and Upadhyay, 2021; Zhang et al., 2020; Ang et al., 2022; Duong et al., 2022; Santana et al., 2022; Maisto et al., 2022; Mattsson et al., 2022; Nabi et al., 2022; Razeghi et al., 2022; Schütze et al., 2022; Debraj and Lavanya, 2023
Sodium Iodide (NaI)	1.55–1.8	Ideal density to recover also high-density polymers. Potential for solution recycling and reuse.	Expensive. High toxicity and hazard. May darken cellulose filters, hindering visual sorting. Hygroscopic and unstable in solution.	Claessens et al., 2013; Nuelle et al., 2014; Van Cauwenberghe et al., 2015; Coppock et al., 2017; Crichton et al., 2017; Kedzierski et al., 2017; Ling et al., 2017; Quinn et al., 2017; Willis et al., 2017; Frias et al., 2018; Han et al., 2019; Stock et al., 2019; Liu et al., 2020; Mehdinia et al., 2020; Thomas et al., 2020; Bellasi et al., 2021; Cutroneo et al., 2021; Kumar and Sharma, 2021; Nava and Leoni, 2021; Tirkey and Upadhyay, 2021; Zhang et al., 2021; Ang et al., 2022; Duong et al., 2022; Santana et al., 2022; Maisto et al., 2022; Mattsson et al., 2022; Nabi et al., 2022; Razeghi et al., 2022; Debraj and Lavanya, 2023
Sodium Polytungstate ($3Na_2WO_4{\cdot}9WO_3{\cdot}H2O$)	1.4–1.8	Ideal density to recover also high-density polymers. Potential for solution recycling and reuse. Low toxicity.	Expensive. May crystallize during evaporation.	Gregory and Johnston, 1987; Torresan, 1987, Krukowski, 1988; Skipp, 1993; Corcoran et al., 2009; Corcoran et al., 2015; Martin et al., 2017; Dean et al., 2018; Helbig and Pitt, 2018; Enders et al., 2019; Stock et al., 2019; Turner et al., 2019; Cunningham et al., 2020; Thomas et al., 2020; Cutroneo et al., 2021; Eskola et al., 2021; Razeghi et al., 2022; Weber and Kerpen, 2023
Sodium Tungstate Dihidrate ($Na_2WO_4{\cdot}2H_20$)	1.4	Relatively cheap. Low toxicity.	Low density to recover high-density polymers.	Frias et al., 2018; Pagter et al., 2018; Kanhai et al., 2019; Stock et al., 2019; Pagter et al., 2020; Thomas et al., 2020; Cutroneo et al., 2021; Tsering et al., 2022
Zinc Bromide ($ZnBr_2$)	1.7	Ideal density to recover also high-density polymers. Potential for solution recycling and reuse.	Expensive. High toxicity. Corrosive.	Frias et al., 2018; Quinn et al., 2017; Thomas et al., 2020; Wu et al., 2020; Cutroneo et al., 2021; Eskola et al., 2021; Tirkey and Upadhyay, 2021; Debraj and Lavanya, 2023
Zinc Chloride ($ZnCl_2$)	1.5–1.8	Ideal density to recover also high-density polymers. Potential for solution recycling and reuse.	Expensive. High toxicity. Corrosive. May cause alteration and degradation of MPs. May cause foaming in organic-rich samples.	Van Cauwenberghe et al., 2015; Horton et al., 2017; Zobkov and Esiukova, 2017; Frias et al., 2018; Lo et al., 2018; Stock et al., 2019; Zheng et al., 2019; Konechnaya et al., 2020; Liu et al., 2020; Rodrigues et al., 2020; Thomas et al.,

(*continued on next page*)

The collected sediments were stored in aluminium foils. Before artificial sample preparation, the sediments were dried at 50 °C, sieved, and separated into two grain size ranges: particles smaller than 63 μm (mud) and particles ranging from 63 and 250 μm (very fine to fine sand), for composition analysis. The 63 to 250 μm fraction was analysed through petrographic analysis (composition dominated by quartz, with minor amounts of feldspars, volcanic grains, and mica). In contrast, the fraction of particles smaller than 63 μm was examined using X-ray powder diffraction (composed of clay minerals, quartz, albite, chlorite, and orthoclase). Three different sediment mixtures were prepared: i) Mixture A (100 % mud), with all particles smaller than 63 μm, ii) Mixture B (50 % mud and 50 % sand) containing 50 % of particles smaller than 63 μm and 50 % of particles ranging from 63 to 250 μm, and iii) Mixture C (100 % sand) with particle sizes between 63 and 250 μm.

2.2. MPs preparation

MPs were obtained by manual cutting under a magnifying glass with precision scissors, segments of a PET (density of 1.38 g cm^{-3}) blue drawstring. The resulting fibres were 100 to 500 μm long and showed a diameter of 17 μm.

2.3. Artificial sediment samples

Sediment samples used during the experiments were generated by introducing 50 MP items into 20 g (dry weight) of plastic-free sediments. The addition of 50 MP particles into the samples was carried out under the microscope. Within the microscope field, individual PET fibres, manually selected to be between 100 and 500 μm in size, were placed on a drop of deionised water sited on a slide to prevent their dispersion during the transfer procedure. Subsequently, aided by additional deionised water, the fibres were carefully transferred into 300 mL glass beakers containing 20 g of sediment to create the study artificial sediments. A total of 20 samples for each mixture were created, for a total of 60 samples.

2.4. The sodium polytungstate solution and the separation procedure

A saturated solution of 3.1 g cm^{-3} of SPT was prepared by dissolving the salt granules in deionised water using a magnetic stirrer plate. Afterwards, deionised water was added until the required density of 1.6 g cm^{-3} was reached. This density was selected because it falls above that of plastics yet below sediments, enabling the principle of density separation to operate effectively. The solution was then filtered through a vacuum pump using a 0.45 μm cellulose filter (GVS filter technology) and stored in a glass beaker.

The density separation process began by adding and mixing 100 mL of the SPT solution within the beakers containing the artificial samples. Following the complete settling of sediments ($\geq$ 12 h.), the suspended particulate matter – lighter than the SPT solution and containing MPs – was collected using a pipette. The entire surface of the liquid was sampled uniformly, ensuring no preferential collection, and the solution was transferred entirely to cellulose acetate filters with 0.45 μm pores (GVS filter technology). The filters were placed in a filtration funnel to support the filtration. Following Frias et al. (2018), the top of the funnel was covered with aluminium foil to avoid contamination during the filtration. Additionally, all instrumentation (e.g., the pipette, the walls of the filtration device, the beakers) used for the procedure was rinsed with pre-filtered water before the procedure, to prevent contamination, and after the procedure to ensure that all fibres were fully recovered. The filters were placed in aluminium containers and covered with aluminium foil until microscope observation.

To evaluate the recovery rates, PET fibres trapped in the stored filters were counted using a stereo microscope. The recovery rate for different samples is expressed as a percentage of the recovered fibres compared to the 50 items added to each sample to create the artificial mixture.

2.5. The four experimental setups

This section details the methodologies employed for the extraction procedures conducted with the prepared artificial sediment samples. A total of 15 series of experiments were conducted across four distinct procedures (Fig. 1), corresponding to five experiments for each of the three mixtures. The transition from one procedure to another occurred after evaluating the results of the previous procedure to enhance the recovery rate. The detailed reasons for the success or failure of each procedure, and thus the shift to the next one, will be thoroughly discussed in Section 3.

Procedure 1 began by adding and mixing the SPT solution into the samples contained in the beakers. The sediments were then allowed to settle for 12 h., after which the supernatant was filtered as described by Masura et al. (2015) and Frias et al. (2018) protocols. In Procedure 2, beakers containing sediments and the SPT solution underwent a 5-min ultrasonic bath treatment to separate possible MP-sediment aggregates (Wang et al., 2018; Zhang et al., 2018; Alvim et al., 2021). The duration of the sonication step and the used frequency (40 K Hz) were chosen to avoid possible degradation of the MP particles (von der Esch et al., 2020; Goli and Singh, 2023). Following sonication, the samples were left undisturbed for 12 h.

to allow complete sediment settling, after which the supernatant was filtered. Procedure 3 involved repeating the steps of Procedure 2 twice for each sample, as a second treatment may aid in the complete extraction of all MP particles (Debraj and Lavanya, 2023). The steps were carried out in the following sequence. SPT solution was added to the samples, followed by a 5-min sonication. Once the sediments had fully settled, the supernatant was filtered. An additional 100 mL of SPT solution was then added to the remaining sediments in the beakers, and the process – sonication, settling, and filtration – was repeated. Due to the abundance of fine-grained sediments in mixture B samples, after the initial sonication phase, the suspension was transferred to a new beaker. The settled sediments were left in the original one to maximize the extraction of MPs from the coarser portion of the sample. Procedure 4 does not include sonication and involves placing the glass beakers with the sediments and the SPT solution on a stirring plate for a total of 3 h. with a progressively lowering rotation velocity (300 rpm, 1st h.; 200 rpm, 2nd h.; 100 rpm, 3rd h.) to allow sediments to settle out progressively according to their grain size. After the 12 h. for sediment settling, the extraction of MPs was carried out. To ensure that there was no degradation or further fragmentation of the particles during the magnetic stirring process, PET fibres were carefully observed both before

Table. 1 (*continued*)

Salt	Density ($g \cdot cm^{-3}$)	Strengths	Weaknesses	References
				2020; Wu et al., 2020; Bellasi et al., 2021; Cutroneo et al., 2021; Esiukova et al., 2021; Eskola et al., 2021; Radford and Zapata-Restrepo, 2021; Tirkey and Upadhyay, 2021; Ang et al., 2022; Duong et al., 2022; Mattsson et al., 2022; Nabi et al., 2022; Razeghi et al., 2022; Schrank et al., 2022; Debraj and Lavanya, 2023

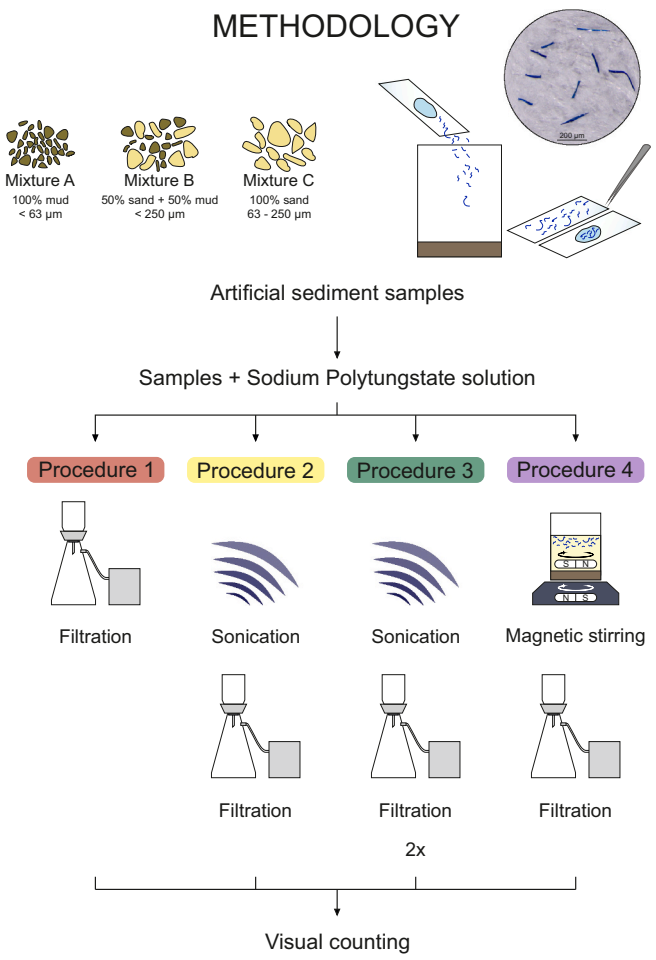


Fig. 1. Schematic diagram illustrating the four separation procedures. The flowchart progresses as follows: first, three artificial sediment mixtures are created by incorporating PET fibres into plastic-free sediments. Sodium Polytungstate solution is then added to each sample to conduct density separation. After completing the four separation procedures, MPs are quantified by observing the filters under a stereomicroscope.

and after the procedure.

3. Results and discussion

Results obtained from applying the four procedures to the three artificial mixtures are summarized in Fig. 2. In detail, the overall recovery rate across the three different sediment mixtures was 46 % ± 10 %, 49 % ± 9 %, 63 % ± 8 %, and 92 % ± 2 % from Procedure 1 to 4, respectively. Notably, the graphical representation shows a significant reduction in the interquartile range (IQR) from Procedures 1 to 4. The extraction of MPs consistently exhibited a higher efficacy in Mixture C for all procedures, reaching values of 98 % ± 1 % in Procedure 4, compared to Mixture A and B samples. However, the improvement from Procedures 1 to 4 is particularly evident in sediment mixtures A and B. Starting from values of 24 % ± 7 % (Mixture A) and 20 % ± 8 % (Mixture B) in Procedure 1, they have progressed to values of 86 % ± 2 % (Mixture A) and 92 % ± 4 % (Mixture B) in Procedure 4.

These results clearly show differences in recovery rates among the four procedures, as well as variations among the three artificial sediment mixtures. For instance, extracting MPs was found to be more straightforward in samples containing sand-sized sediments than in mud-rich samples.

The elevated retrieval of MPs reached in all four procedures from Mixture C samples can be attributed to the substantial contrast in settling velocity between MPs – particularly particles exhibiting irregular shapes, such as fibres – and sand particles (Waldschläger and Schüttrumpf, 2019; Wang et al., 2021a, 2021b; Qian et al., 2024). The rapid settling of sand grains compared to the one of MPs and the considerable spacing between sandy sediment particles prevent the formation of a sediment seal and MPs can easily rise to the liquid surface. Thus, optimal recovery of MPs when working with mud-free sandy sediments (e.g. wave-reworked deposits) can be achieved through an easy procedure involving only a few steps. Conversely, a more complex procedure is required to ensure a good recovery in samples containing sediments characterised by fine-grained particles (e.g., floodplains and tidal flat deposits). Results obtained from the extraction of MPs from mud-rich sediments were, in fact, different. This is mainly due to the dynamic interplays between MPs and sediment particles with a grain size lower than 63 µm. Therefore, when extracting MPs from sediments through density separation, it is necessary to consider the particle size of the sediments. Sediments can impact the behaviour of MPs, and it is well known that the interactions between MPs and fine-grained sediment particles, such as clay, have effects on the transport and spatial dispersion of MPs (Besseling et al., 2017; Kooi et al., 2018; Del Domercq et al., 2022). These fine-grained and cohesive sediments can adhere to the surfaces of MPs, creating aggregates characterised by an increased density and influencing their hydrodynamic behaviour (Alimi et al., 2018; Li et al., 2019; Wu et al., 2020; de Haan et al., 2019; Leiser et al., 2020). The formation of these aggregates is linked to the frequency and energy of particle interactions (Li et al., 2018; Wang et al., 2021a, 2021b), factors that are dependent on sediment concentration and the prevailing hydrodynamic conditions. Additionally, the electrostatic charge of surfaces and the salinity of the fluid play central roles, with higher salinity correlating to an increased affinity to the formation of MPs-sediment aggregates (Andersen et al., 2021; Laursen et al., 2022; Laursen et al., 2023). Hence, in the presence of suspended sediments, the formation of MPs-sediment aggregates modifies the hydrodynamic behaviour of MP particles that, in the form of aggregates, tend to settle and accumulate within sediments (Li et al., 2019) preventing an efficient recovery. Additionally, suspended cohesive sediments can create a cap, preventing MPs from rising to the surface (Kane and Clare, 2019; Pohl et al., 2020; Li et al., 2021). Considering the recovery of MPs from sand has been high since Procedure 1, the introduction of Procedures 2 to 4 became necessary to increase the recovery when extracting MPs from the two other sediment mixtures. In Procedure 2, a sonication step was introduced as ultrasounds are commonly employed to remove cohesive particles adhered to MP surfaces (Wang et al., 2018; Zhang et al., 2018; Alvim et al., 2021). In this procedure, the low frequency, and the short sonication time, mandatory to prevent MP degradation, were insufficient to disaggregate flocs and the recovery remained low. In Procedure 3, a further step in attempting an increased recovery was repeating the extraction procedure twice per sample while maintaining the use of ultrasounds for both times. The addition of one or more separation steps is often employed in literature mainly when using a saline solution such as NaCl where the low density of the liquid does not allow the recovery of all MP particles at once (Debraj and Lavanya, 2023). However, the recovery improved only in Mixture B samples, while it remained low for Mixture A ones. The enhancement in recovery from Mixture B samples is primarily attributed to the subdivision between the sand and mud fractions of Mixture B samples. Performing the separation procedure individually for the two sample portions increased the extraction of MPs from the coarser portion. Results from the application of Procedures 2 and 3 suggest that interaction between MP and cohesive sediments persists despite sonication and repeated filtration process. Since increasing sonication frequency/duration may result in MP fragmentation and, along with repeated filtration, in time-consuming procedures, Procedure 4 has been developed to prevent MP aggregation with sediments. In Procedure 4, placing glass beakers containing sediments and the SPT solution on a stirring plate allows MPs to rise to the liquid column. Meanwhile, a gradual reduction in stirring speed enables

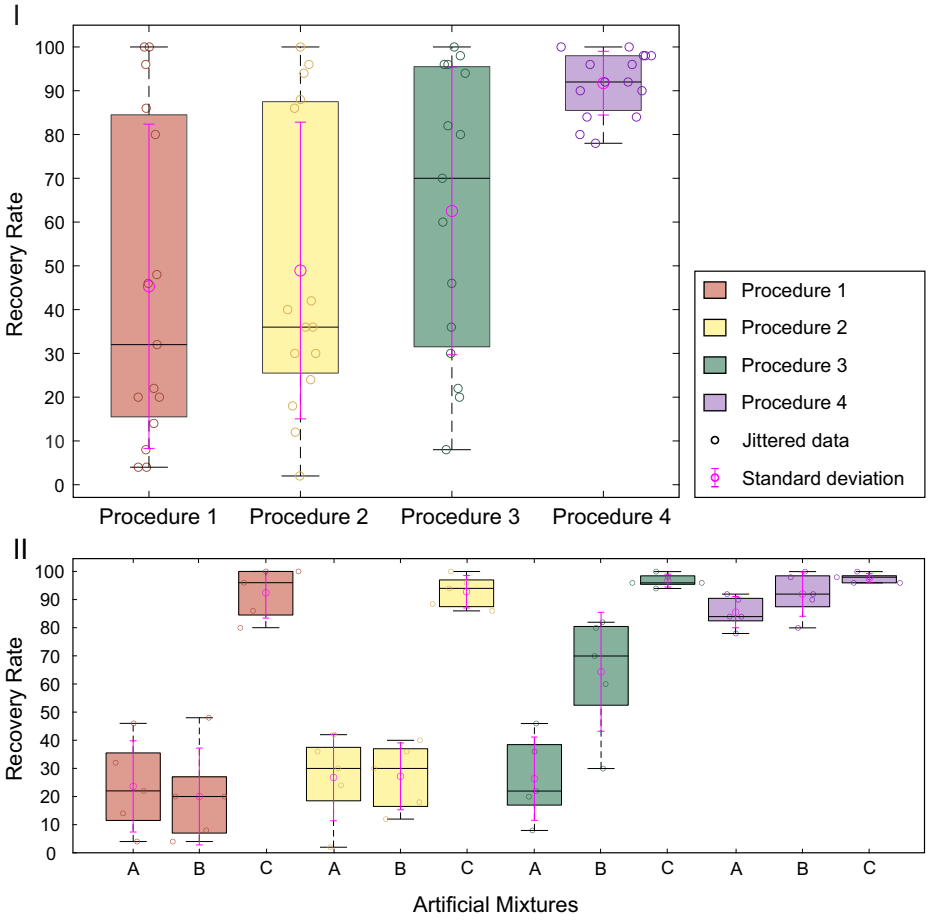


Fig. 2. Boxplot chart illustrating the overall recovery variability across the four separation procedures (I). Boxplot chart displaying the recoveries from the three artificial mixtures (A, B, and C) for the four separation procedures (II).

cohesive sediment particles to settle gradually without forming aggregates. Through this mechanism, cohesive sediments were no longer able to massively push MPs towards the bottom and this allowed MP particles to reach the liquid surface.

The results of this study indicate that based on the strengths and limitations of salt solutions commonly used in the literature for MP extraction from sediments, a SPT solution with a density of 1.6 g cm^{-3} proves to be among the best choices. However, precautions must be taken based on the type and particle size of the sediment from which microplastics are being extracted. A simple filtration (Procedure 1 of this study) using SPT would be an efficient and time-saving procedure to be used for MPs separation from sediments accumulated in mud-free depositional settings, like wave-reworked environments (e.g. beaches, shorefaces), aeolian settings (e.g., shoreline dunes, desert dunes) or (in some cases) alluvial channels. When processing sediments accumulated in environments such as lagoons, floodplains, or other low-energy settings that allow for mud accumulation, additional precautions are necessary. These environments include areas below the wave base (marine or lacustrine) or river channels during the waning phase of a flood. In such cases, modifications to the procedure are required to ensure the efficient separation of microplastics from fine-grained sediments. Therefore, for sediments with a significant amount of mud, it is recommended to use Procedure 4 from this study to achieve optimal MP extraction. This method addresses the challenges associated with fine-grained particles and improves the effectiveness of the extraction process in these samples.

4. Conclusions and future directions

The absence of standardised processing methods in extracting MP particles from sediments can compromise comparability between different studies. Density separation is a predominant method employed for extracting MPs from natural sediments, although multiple procedures and tools are used. The two main outcomes of this study are that an SPT saline solution with a density of 1.6 g cm^{-3} is one of the most effective options for MP fibre extraction from environmental sediment samples. However, particular care must be taken to account for the sediment type and particle size of the samples during extraction. In detail:

- MP fibre particles can be efficiently recovered from mud-free sandy sediments by applying an easy procedure involving exclusively solution filtration.
- Achieving an efficient extraction of fibres from mud-rich sand and fully muddy sediments can be accomplished by applying a procedure that involves placing the samples with the SPT saline solution on a stirring plate progressively lowering the rotation velocity.
- The high retrieval efficiency achievable through the extraction of MP fibres from sediments widespread in most of the depositional environments, coupled with the characteristics of the SPT solution – such as its adjustable density, low toxicity, and capacity for repeated recovery and reuse – make it an excellent solution for extracting MPfibres from environmental sediment samples.

However, additional tests are necessary to address the limitations of

the procedure. Future research should consider factors like the varying shapes of MP particles, which may interact and behave differently with sediments, the diversity of polymer types, and, crucially, the addition of a step to remove organic matter from samples without affecting particle recovery. This will enable the reliable analysis of more complex environmental samples, such as floodplains or riverine sediments while ensuring high recovery rates.

CRediT authorship contribution statement

Francesca Uguagliati: Writing – original draft, Investigation, Formal analysis, Data curation, Conceptualization. **Massimiliano Zattin:** Writing – review & editing, Project administration, Methodology, Data curation, Conceptualization. **Kryss Waldschläger:** Writing – review & editing, Conceptualization. **Massimiliano Ghinassi:** Writing – review & editing, Resources, Project administration, Data curation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This study was supported by Enel Green Power, project title: “Riconoscimento, quantificazione e modelli di distribuzione di microplastiche in sedimenti fluviali”. Associate Editor D. Barcelò and two anonymous reviewers are kindly acknowledged for their constructive comments.

Data availability

Data will be made available on request.

References

Abidli, S., Antunes, J.C., Ferreira, J.L., Lahbib, Y., Sobral, P., Trigui El Menif, N., 2018. Microplastics in sediments from the littoral zone of the north Tunisian coast (Mediterranean Sea). Estuar. Coast. Shelf Sci. 205, 1–9. https://doi.org/10.1016/j.ecss.2018.03.006.

Adams, J.K., Dean, B.Y., Athey, S.N., Jantunen, L.M., Bernstein, S., Stern, G., Diamond, M.L., Finkelstein, S.A., 2021. Anthropogenic particles (including microfibers and microplastics) in marine sediments of the Canadian Arctic. Sci. Total Environ. 784, 147155. https://doi.org/10.1016/j.scitotenv.2021.147155.

Akanyange, S.N., Zhang, Y., Zhao, X., Adom-Asamoah, G., Ature, A.-R.A., Anning, C., Tianpeng, C., Zhao, H., Lyu, X., Crittenden, J.C., 2022. A holistic assessment of microplastic ubiquitousness: pathway for source identification in the environment. Sustainable Production and Consumption 33, 113–145. https://doi.org/10.1016/j.spc.2022.06.020.

Alimi, O.S., Farner Budarz, J., Hernandez, L.M., Tufenkji, N., 2018. Microplastics and Nanoplastics in aquatic environments: aggregation, deposition, and enhanced contaminant transport. Environ. Sci. Technol. 52 (4), 1704–1724. https://doi.org/10.1021/acs.est.7b05559.

Alvim, C., Bes-Piá, M.A., Mendoza-Roca, J.A., 2021. An innovative approach to the application of ultrasounds to remove polyethylene microspheres from activated sludge. Sep. Purif. Technol. 264, 118429. https://doi.org/10.1016/j.seppur.2021.118429.

Andersen, T.J., Rominikan, S., Olsen, I.S., Skinnebach, K.H., Fruergaard, M., 2021. Flocculation of PVC microplastic and fine-grained cohesive sediment at environmentally realistic concentrations. Biol. Bull. 240, 42–51. https://doi.org/10.1086/712929.

Ang, A.L.T., Jose, C.M., Del Rosario, C.I.P., Orrin, L.T.U., Garcia, J., 2022. Recent advances on density separation techniques for microplastic recovery from sediments. Sinaya: A Philippine Journal for Senior High School Teachers and Students 1: Iss. 2, Article 1. https://doi.org/10.59588/3027-9283.1010.

Barnes, D.K.A., Galgani, F., Thompson, R.C., Barlaz, M., 2009. Accumulation and fragmentation of plastic debris in global environments. Philos. Trans. R. Soc. B 364, 1985–1998. https://doi.org/10.1098/rstb.2008.0205.

Bayo, J., Rojo, D., Olmos, S., 2019. Abundance, morphology and chemical composition of microplastics in sand and sediments from a protected coastal area: the mar Menor lagoon (SE Spain). Environ. Pollut. 252, 1357–1366. https://doi.org/10.1016/j.envpol.2019.06.024.

Bellasi, A., Binda, G., Pozzi, A., Boldrocchi, G., Bettinetti, R., 2021. The extraction of microplastics from sediments: an overview of existing methods and the proposal of a new and green alternative. Chemosphere 278, 130357. https://doi.org/10.1016/j.chemosphere.2021.130357.

Bellasi, A., Binda, G., Pozzi, A., Galafassi, S., Volta, P., Bettinetti, R., 2020. Microplastic contamination in freshwater environments: a review, focusing on interactions with sediments and benthic organisms. Environments 7 (4), 30. https://doi.org/10.3390/environments7040030.

Besseling, E., Quik, J.T.K., Sun, M., Koelmans, A.A., 2017. Fate of nano- and microplastic in freshwater systems: a modeling study. Environ. Pollut. 220, 540–548. https://doi.org/10.1016/j.envpol.2016.10.001.

Blair, R.M., Waldron, S., Gauchotte-Lindsay, C., 2019. Average daily flow of microplastics through a tertiary wastewater treatment plant over a ten-month period. Water Res. 163, 114909.

Brennecke, D., Duarte, B., Paiva, F., Caçador, I., Canning-Clode, J., 2016. Microplastics as vector for heavy metal contamination from the marine environment. Estuar. Coast. Shelf Sci. 178, 189–195. https://doi.org/10.1016/j.ecss.2015.12.003.

Bridson, J.H., Abbel, R., Smith, D.A., Northcott, G.L., Gaw, S., 2023. Release of additives and non-intentionally added substances from microplastics under environmentally relevant conditions. Environmental Advances 12, 100359. https://doi.org/10.1016/j.envadv.2023.100359.

Browne, M.A., Crump, P., Niven, S.J., Teuten, E., Tonkin, A., Galloway, T., Thompson, R., 2011. Accumulation of microplastic on shorelines worldwide: sources and sinks. Environ. Sci. Technol. 45, 9175–9179. https://doi.org/10.1021/es201811s.

Burns, E.E., Boxall, A.B.A., 2018. Microplastics in the aquatic environment: evidence for or against adverse impacts and major knowledge gaps. Environ. Toxicol. Chem. 37, 2776–2796. https://doi.org/10.1002/etc.4268.

Chouchene, K., Prata, J.C., da Costa, J., Duarte, A.C., Rocha-Santos, T., Ksibi, M., 2021. Microplastics on Barra beach sediments in Aveiro. Portugal. Marine Pollution Bulletin 167, 112264. https://doi.org/10.1016/j.marpolbul.2021.112264.

Chu, J., Griesmann, U., Wang, Q., Soons, J.A., Benck, E.C., 2010. Deformation-free form error measurement of thin, plane-parallel optics floated on a heavy liquid. Appl. Opt., AO 49, 1849–1858. doi:https://doi.org/10.1364/AO.49.001849.

Claessens, M., Van Cauwenberghe, L., Vandegehuchte, M.B., Janssen, C.R., 2013. New techniques for the detection of microplastics in sediments and field collected organisms. Mar. Pollut. Bull. 70, 227–233. https://doi.org/10.1016/j.marpolbul.2013.03.009.

Cole, M., Lindeque, P., Halsband, C., Galloway, T.S., 2011. Microplastics as contaminants in the marine environment: a review. Mar. Pollut. Bull. 62, 2588–2597. https://doi.org/10.1016/j.marpolbul.2011.09.025.

Coppock, R.L., Cole, M., Lindeque, P.K., Queirós, A.M., Galloway, T.S., 2017. A small-scale, portable method for extracting microplastics from marine sediments. Environ. Pollut. 230, 829–837. https://doi.org/10.1016/j.envpol.2017.07.017.

Corcoran, P.L., Biesinger, M.C., Grifi, M., 2009. Plastics and beaches: a degrading relationship. Mar. Pollut. Bull. 58, 80–84. https://doi.org/10.1016/j.marpolbul.2008.08.022.

Corcoran, P.L., Norris, T., Ceccanese, T., Walzak, M.J., Helm, P.A., Marvin, C.H., 2015. Hidden plastics of Lake Ontario, Canada and their potential preservation in the sediment record. Environ. Pollut. 204, 17–25. https://doi.org/10.1016/j.envpol.2015.04.009.

Crichton, E.M., Noël, M., Gies, E.A., Ross, P.S., 2017. A novel, density-independent and FTIR-compatible approach for the rapid extraction of microplastics from aquatic sediments. Anal. Methods 9, 1419–1428. https://doi.org/10.1039/C6AY02733D.

Cunningham, E.M., Ehlers, S.M., Dick, J.T.A., Sigwart, J.D., Linse, K., Dick, J.J., Kiriakoulakis, K., 2020. High abundances of microplastic pollution in Deep-Sea sediments: evidence from Antarctica and the Southern Ocean. Environ. Sci. Technol. 54, 13661–13671. https://doi.org/10.1021/acs.est.0c03441.

Cutroneo, L., Reboa, A., Geneselli, I., Capello, M., 2021. Considerations on salts used for density separation in the extraction of microplastics from sediments. Mar. Pollut. Bull. 166, 112216. https://doi.org/10.1016/j.marpolbul.2021.112216.

Darabi, M., Majeed, H., Diehl, A., Norton, J., Zhang, Y., 2021. A review of microplastics in aquatic sediments: occurrence, fate, transport, and ecological impact. Curr. Pollut. Rep. 7, 40–53. https://doi.org/10.1007/s40726-020-00171-3.

De Arbeloa, N.P., Marzadri, A., 2024. Modeling the transport of microplastics along river networks. Sci. Total Environ. 911, 168227. https://doi.org/10.1016/j.scitotenv.2023.168227.

de Haan, W.P., Sanchez-Vidal, A., Canals, M., 2019. Floating microplastics and aggregate formation in the Western Mediterranean Sea. Mar. Pollut. Bull. 140, 523–535. https://doi.org/10.1016/j.marpolbul.2019.01.053.

Dean, B.Y., Corcoran, P.L., Helm, P.A., 2018. Factors influencing microplastic abundances in nearshore, tributary and beach sediments along the Ontario shoreline of Lake Erie. J. Great Lakes Res. 44, 1002–1009. https://doi.org/10.1016/j.jglr.2018.07.014.

Debraj, D., Lavanya, M., 2023. Microplastics everywhere: a review on existing methods of extraction. Sci. Total Environ. 893, 164878. https://doi.org/10.1016/j.scitotenv.2023.164878.

Del Domercq, P., Praetorius, A., MacLeod, M., 2022. The full multi: an open-source framework for modelling the transport and fate of nano- and microplastics in aquatic systems. Environ. Model Softw. 148, 105291. https://doi.org/10.1016/j.envsoft.2021.105291.

Duong, T.T., Le, P.T., Nguyen, T.N.H., Hoang, T.Q., Ngo, H.M., Doan, T.O., Le, T.P.Q., Bui, H.T., Bui, M.H., Trinh, V.T., Nguyen, T.L., Da Le, N., Vu, T.M., Tran, T.K.C., Ho, T.C., Phuong, N.N., Strady, E., 2022. Selection of a density separation solution to study microplastics in tropical riverine sediment. Environ. Monit. Assess. 194, 65. https://doi.org/10.1007/s10661-021-09664-0.

Enders, K., Käppler, A., Biniasch, O., Feldens, P., Stollberg, N., Lange, X., Fischer, D., Eichhorn, K.-J., Pollehne, F., Oberbeckmann, S., Labrenz, M., 2019. Tracing microplastics in aquatic environments based on sediment analogies. Sci. Rep. 9, 15207. https://doi.org/10.1038/s41598-019-50508-2.

Esiukova, E.E., Lobchuk, O.I., Volodina, A.A., Chubarenko, I.P., 2021. Marine macrophytes retain microplastics. Mar. Pollut. Bull. 171, 112738. https://doi.org/10.1016/j.marpolbul.2021.112738.

Eskola, T., Kontio, R., Lunkka, J.P., 2021. Comparison between modified LST Fastfloat and conventional HF methods for pollen preparation in highly minerogenic sediments. Bull. Geol. Soc. Finl. 93, 5–18. https://doi.org/10.17741/bgsf/93.1.001.

Ferreira, M., Thompson, J., Paris, A., Rohindra, D., Rico, C., 2020. Presence of microplastics in water, sediments and fish species in an urban coastal environment of Fiji, a Pacific small island developing state. Mar. Pollut. Bull. 153, 110991. https://doi.org/10.1016/j.marpolbul.2020.110991.

Filgueiras, A.V., Gago, J., Campillo, J.A., León, V.M., 2019. Microplastic distribution in surface sediments along the Spanish Mediterranean continental shelf. Environ. Sci. Pollut. Res. 26, 21264–21273. https://doi.org/10.1007/s11356-019-05341-5.

Frias, J.P.G.L., Pagter, E., Nash, R., O'Connor, I., Carretero, O., Filgueiras, A., Viñas, L., J. Gago, Antunes, J.C., Bessa, F., Sobral, P., Goruppi, A., Tirelli, V., Pedrotti, M.L., Suaria, G., Aliani, S., Lopes, C., Raimundo, J., Caetano, M., Palazzo, L., Lucia, G.A.D., Camedda, A., Muniategui, S., Grueiro, G., Fernandez, V., Andrade, J., Dris, R., Laforsch, C., Scholtz-Bottcher, B., Gerdts, G., 2018. Standardised protocol for monitoring microplastics in sediments. Doi:10.13140/RG.2.2.36256.89601/1.

Gazal, A.A., Gheewala, S.H., 2020. Plastics, microplastics and other polymer materials - A threat to the environment. Journal of Sustainable Energy & Environment 11, 113–122.

Ghinassi, M., Fidolini, F., Magi, M., Sagri, M., 2013. Depositional environments of the Plio-Pleistocene upper Valdarno Basin (Tuscany, Italy). IJG 132 (2013) f.1. doi: https://doi.org/10.3301/IJG.2012.07.

Ghosh, Shampa, Sinha, J.K., Ghosh, Soumya, Vashisth, K., Han, S., Bhaskar, R., 2023. Microplastics as an emerging threat to the global environment and human health. Sustainability 15, 10821. https://doi.org/10.3390/su151410821.

Godoy, V., Blázquez, G., Calero, M., Quesada, L., Martín-Lara, M.A., 2019. The potential of microplastics as carriers of metals. Environ. Pollut. 255, 113363. https://doi.org/10.1016/j.envpol.2019.113363.

Goli, V.S.N.S., Singh, D.N., 2023. Effect of ultrasonication conditions on polyethylene microplastics sourced from landfills: a precursor study to establish guidelines for their extraction from environmental matrices. J. Hazard. Mater. 459, 132230. https://doi.org/10.1016/j.jhazmat.2023.132230.

Gregory, M.R., Johnston, K.A., 1987. A nontoxic substitute for hazardous heavy liquids—aqueous sodium polytungstate ($3Na_2WO_4.9WO_3.H_2O$) solution (Note). N. Z. J. Geol. Geophys. 30, 317–320. doi:https://doi.org/10.1080/00288306.1987.10552626.

Halbach, M., Baensch, C., Dirksen, S., Scholz-Böttcher, B.M., 2021. Microplastic extraction from sediments established? – a critical evaluation from a trace recovery experiment with a custom-made density separator. Anal. Methods 13, 5299–5308. https://doi.org/10.1039/D1AY00983D.

Hamid, S.F., Bhatti, M.S., Anuar, Norkhairiyah, Anuar, Norkhairah, Mohan, P., Periathamby, A., 2018. Worldwide distribution and abundance of microplastic: how dire is the situation? Waste Manag. Res. 36, 873–897. https://doi.org/10.1177/0734242X18785730.

Han, N., Zhao, Q., Ao, H., Hu, H., Wu, C., 2022. Horizontal transport of macro- and microplastics on soil surface by rainfall induced surface runoff as affected by vegetations. Sci. Total Environ. 831, 154989. https://doi.org/10.1016/j.scitotenv.2022.154989.

Han, X., Lu, X., Vogt, R.D., 2019. An optimized density-based approach for extracting microplastics from soil and sediment samples. Environ. Pollut. 254, 113009. https://doi.org/10.1016/j.envpol.2019.113009.

Hanvey, J.S., Lewis, P.J., Lavers, J.L., Crosbie, N.D., Pozo, K., Clarke, B.O., 2017. A review of analytical techniques for quantifying microplastics in sediments. Anal. Methods 9, 1369–1383. https://doi.org/10.1039/C6AY02707E.

He, B., Smith, M., Egodawatta, P., Ayoko, G.A., Rintoul, L., Goonetilleke, A., 2021. Dispersal and transport of microplastics in river sediments. Environ. Pollut. 279, 116884. https://doi.org/10.1016/j.envpol.2021.116884.

Helbig, P., Pitt, C., D., 2018. Sodium polytungstate as gravity separating fluid for polymeric blasting media evaluation. Anal. Methods 10, 3039–3042. https://doi.org/10.1039/C8AY00946E.

Horton, A.A., Svendsen, C., Williams, R.J., Spurgeon, D.J., Lahive, E., 2017. Large microplastic particles in sediments of tributaries of the river Thames, UK – abundance, sources and methods for effective quantification. Mar. Pollut. Bull. 114, 218–226. https://doi.org/10.1016/j.marpolbul.2016.09.004.

Jiang, J.-Q., 2018. Occurrence of microplastics and its pollution in the environment: a review. Sustainable Production and Consumption 13, 16–23. https://doi.org/10.1016/j.spc.2017.11.003.

Kane, I.A., Clare, M.A., 2019. Dispersion, accumulation, and the ultimate fate of microplastics in deep-marine environments: a review and future directions. Front. Earth Sci. 7, 80. https://doi.org/10.3389/feart.2019.00080.

Kanhai, L.D.K., Johansson, C., Frias, J.P.G.L., Gardfeldt, K., Thompson, R.C., O'Connor, I., 2019. Deep sea sediments of the Arctic Central Basin: a potential sink for microplastics. Deep-Sea Res. I Oceanogr. Res. Pap. 145, 137–142. https://doi.org/10.1016/j.dsr.2019.03.003.

Kedzierski, M., Le Tilly, V., César, G., Sire, O., Bruzaud, S., 2017. Efficient microplastics extraction from sand. A cost effective methodology based on sodium iodide recycling. Mar. Pollut. Bull. 115, 120–129. https://doi.org/10.1016/j.marpolbul.2016.12.002.

Konechnaya, O., Lüchtrath, S., Dsikowitzky, L., Schwarzbauer, J., 2020. Optimized microplastic analysis based on size fractionation, density separation and μ-FTIR. Water Sci. Technol. 81, 834–844. https://doi.org/10.2166/wst.2020.173.

Kooi, M., Besseling, E., Kroeze, C., van Wezel, A.P., Koelmans, A.A., 2018. Modeling the fate and transport of plastic debris in freshwaters: Review and guidance. In: Wagner, M., Lambert, S. (Eds.), Freshwater Microplastics: Emerging Environmental Contaminants? The Handbook of Environmental Chemistry. Springer International Publishing, Cham, pp. 125–152. https://doi.org/10.1007/978-3-319-61615-5_7.

Kooi, M., Koelmans, A.A., 2019. Simplifying microplastic via continuous probability distributions for size, shape, and density. Environ. Sci. Technol. Lett. 6, 551–557. https://doi.org/10.1021/acs.estlett.9b00379.

Kooi, M., Reisser, J., Slat, B., Ferrari, F.F., Schmid, M.S., Cunsolo, S., Brambini, R., Noble, K., Sirks, L.-A., Linders, T.E.W., Schoeneich-Argent, R.I., Koelmans, A.A., 2016. The effect of particle properties on the depth profile of buoyant plastics in the ocean. Sci. Rep. 6, 33882. https://doi.org/10.1038/srep33882.

Krukowski, S.T., 1988. Sodium metatungstate: a new heavy mineral separation medium for the extraction of conodonts from insoluble residues. J. Paleontol. 62 (314–316), 1988.

Kumar Chaudry, A., Sachdeva, P., 2021. Microplastics' origin, distribution, and rising hazard to aquatic organisms and human health: Socio-economic insinuations and managment solutions. Regional Stuides in Marine Science 48, 102018.

Kumar, R., Sharma, P., 2021. Recent developments in extraction, identification, and quantification of microplastics from agricultural soil and groundwater. In: Gupta, P. K., Bharagava, R.N. (Eds.), Fate and Transport of Subsurface Pollutants, Microorganisms for Sustainability. Springer, Singapore, pp. 125–143. https://doi.org/10.1007/978-981-15-6564-9_7.

Kumar, Rakesh, Verma, A., Shome, A., Sinha, R., Sinha, S., Jha, P.K., Kumar, Ritesh, Kumar, P., Shubham S., Das, Sharma, P., Vara Prasad, P.V., 2021. Impacts of plastic pollution on ecosystem services, sustainable development goals, and need to focus on circular economy and policy interventions. Sustainability 13, 9963. https://doi.org/10.3390/su13179963.

Kurzweg, L., Schirrmeister, S., Hauffe, M., Adomat, Y., Socher, M., Harre, K., 2022. Application of electrostatic separation and differential scanning calorimetry for microplastic analysis in river sediments. Frontiers in environmental. Science 10.

Laursen, S.N., Fruergaard, M., Andersen, T.J., 2022. Rapid flocculation and settling of positively buoyant microplastic and fine-grained sediment in natural seawater. Mar. Pollut. Bull. 178, 113619. https://doi.org/10.1016/j.marpolbul.2022.113619.

Laursen, S.N., Fruergaard, M., Dodhia, M.S., Posth, N.R., Rasmussen, M.B., Larsen, M.N., Shilla, Dativa, Shilla, Daniel, Kilawe, J.J., Kizenga, H.J., Andersen, T.J., 2023. Settling of buoyant microplastic in estuaries: the importance of flocculation. Sci. Total Environ. 886, 163976. https://doi.org/10.1016/j.scitotenv.2023.163976.

Lebreton, L., Andrady, A., 2019. Future scenarios of global plastic waste generation and disposal. Palgrave Commun 5, 1–11. https://doi.org/10.1057/s41599-018-0212-7.

Leiser, R., Wu, G.M., Neu, T.R., Wendt-Potthoff, K., 2020. Biofouling, metal sorption and aggregation are related to sinking of microplastics in a stratified reservoir. Water Res., 176 (2020), Article 115748, https://doi.org/10.1016/j.watres.2020.115748.

Li, M., Zhang, X., Yi, K., He, L., Han, P., Tong, M., 2021. Transport and deposition of microplastic particles in saturated porous media: co-effects of clay particles and natural organic matter. Environ. Pollut. 287, 117585. https://doi.org/10.1016/j.envpol.2021.117585.

Li, S., Liu, H., Gao, R., Abdurahman, A., Dai, J., Zeng, F., 2018. Aggregation kinetics of microplastics in aquatic environment: complex roles of electrolytes, pH, and natural organic matter. Environ. Pollut. 237, 126–132. https://doi.org/10.1016/j.envpol.2018.02.042.

Li, Y., Wang, X., Fu, W., Xia, X., Liu, C., Min, J., Zhang, W., Crittenden, J.C., 2019. Interactions between nano/micro plastics and suspended sediment in water: implications on aggregation and settling. Water Res. 161, 486–495. https://doi.org/10.1016/j.watres.2019.06.018.

Ling, S.D., Sinclair, M., Levi, C.J., Reeves, S.E., Edgar, G.J., 2017. Ubiquity of microplastics in coastal seafloor sediments. Mar. Pollut. Bull. 121, 104–110. https://doi.org/10.1016/j.marpolbul.2017.05.038.

Liu, G., Dave, P.H., Kwong, R.W.M., Wu, M., Zhong, H., 2021. Influence of microplastics on the mobility, bioavailability, and toxicity of heavy metals: a review. Bull. Environ. Contam. Toxicol. 107, 710–721. https://doi.org/10.1007/s00128-021-03339-9.

Liu, M., Lu, S., Chen, Y., Cao, C., Bigalke, M., He, D., 2020. Analytical methods for microplastics in environments: Current advances and challenges. In: He, D., Luo, Y. (Eds.), Microplastics in Terrestrial Environments: Emerging Contaminants and Major Challenges, the Handbook of Environmental Chemistry. Springer International Publishing, Cham, pp. 3–24. https://doi.org/10.1007/698_2019_436.

Liu, M., Song, Y., Lu, S., Qiu, R., Hu, J., Li, X., Bigalke, M., Shi, H., He, D., 2019. A method for extracting soil microplastics through circulation of sodium bromide solutions. Sci. Total Environ. 691, 341–347. https://doi.org/10.1016/j.scitotenv.2019.07.144.

Lo, H.S., Xu, X., Wong, C.Y., Cheung, S.G., 2018. Comparisons of microplastic pollution between mudflats and sandy beaches in Hong Kong. Environ. Pollut. 236, 208–217. https://doi.org/10.1016/j.envpol.2018.01.031.

Maisto, M., Ranauda, M.A., Zuzolo, D., Tartaglia, M., Postiglione, A., Prigioniero, A., Falzarano, A., Scarano, P., Sciarrillo, R., Guarino, C., 2022. Influence of sediment texture on HDPE microplastics recovery by density separation. J. Environ. Manag. 317, 115363. https://doi.org/10.1016/j.jenvman.2022.115363.

Martin, J., Lusher, A., Thompson, R.C., Morley, A., 2017. The deposition and accumulation of microplastics in marine sediments and bottom water from the Irish continental shelf. Sci. Rep. 7, 10772. https://doi.org/10.1038/s41598-017-11079-2.

Masura, J., Baker, J., Foster, G., Arthur, C., 2015. Laboratory methods for the analysis of microplastics in the marine environment: recommendations for quantifying

synthetic particles in waters and sediments. (report). NOAA marine debris division. Doi:10.25607/OBP-604.
Mataji, A., Taleshi, M.S., Balimoghaddas, E., 2020. Distribution and characterization of microplastics in surface waters and the southern Caspian Sea coasts sediments. Arch. Environ. Contam. Toxicol. 78, 86–93. https://doi.org/10.1007/s00244-019-00700-2.
Mattsson, K., Ekstrand, E., Granberg, M., Hassellöv, M., Magnusson, K., 2022. Comparison of pre-treatment methods and heavy density liquids to optimize microplastic extraction from natural marine sediments. Sci. Rep. 12, 15459. https://doi.org/10.1038/s41598-022-19623-5.
Mehdinia, A., Dehbandi, R., Hamzehpour, A., Rahnama, R., 2020. Identification of microplastics in the sediments of southern coasts of the Caspian Sea, north of Iran. Environ. Pollut. 258, 113738. https://doi.org/10.1016/j.envpol.2019.113738.
Mu, J., Qu, L., Jin, F., Zhang, S., Fang, C., Ma, X., Zhang, W., Huo, C., Cong, Y., Wang, J., 2019. Abundance and distribution of microplastics in the surface sediments from the northern Bering and Chukchi seas. Environ. Pollut. 245, 122–130. https://doi.org/10.1016/j.envpol.2018.10.097.
Nabi, I., Bacha, A.-U.-R., Zhang, L., 2022. A review on microplastics separation techniques from environmental media. J. Clean. Prod. 337, 130458. https://doi.org/10.1016/j.jclepro.2022.130458.
Nava, V., Leoni, B., 2021. Comparison of different procedures for separating microplastics from sediments. Water 13, 2854. https://doi.org/10.3390/w13202854.
Nuelle, M.-T., Dekiff, J.H., Remy, D., Fries, E., 2014. A new analytical approach for monitoring microplastics in marine sediments. Environ. Pollut. 184, 161–169. https://doi.org/10.1016/j.envpol.2013.07.027.
O'Brine, T., Thompson, R.C., 2010. Degradation of plastic carrier bags in the marine environment. Mar. Pollut. Bull. 60, 2279–2283. https://doi.org/10.1016/j.marpolbul.2010.08.005.
Padervand, M., Lichtfouse, E., Robert, D., Wang, C., 2020. Removal of microplastics from the environment. A review. Environ Chem Lett 18, 807–828. https://doi.org/10.1007/s10311-020-00983-1.
Pagter, E., Frias, J., Kavanagh, F., Nash, R., 2020. Varying levels of microplastics in benthic sediments within a shallow coastal embayment. Estuar. Coast. Shelf Sci. 243, 106915. https://doi.org/10.1016/j.ecss.2020.106915.
Pagter, E., Frias, J., Nash, R., 2018. Microplastics in Galway Bay: a comparison of sampling and separation methods. Mar. Pollut. Bull. 135, 932–940. https://doi.org/10.1016/j.marpolbul.2018.08.013.
Pohl, F., Eggenhuisen, J.T., Kane, I.A., Clare, M.A., 2020. Transport and burial of microplastics in deep-marine sediments by turbidity currents. Environ. Sci. Technol. 54, 4180–4189. https://doi.org/10.1021/acs.est.9b07527.
Qian, S., Qiao, X., Zhang, W., Yu, Z., Dong, S., Feng, J., 2024. Machine learning-based prediction for settling velocity of microplastics with various shapes. Water Res. 249, 121001. https://doi.org/10.1016/j.watres.2023.121001.
Quinn, B., Murphy, F., Ewins, C., 2017. Validation of density separation for the rapid recovery of microplastics from sediment. Anal. Methods 9, 1491–1498. https://doi.org/10.1039/C6AY02542K.
Radford, F., M. Zapata-Restrepo, L., A. Horton, A., D. Hudson, M., J. Shaw, P., D. Williams, I., 2021. Developing a systematic method for extraction of microplastics in soils. Anal. Methods 13, 1695–1705. doi:https://doi.org/10.1039/D0AY02086A.
Razeghi, N., Hamidian, A.H., Mirzajani, A., Abbasi, S., Wu, C., Zhang, Y., Yang, M., 2022. Sample preparation methods for the analysis of microplastics in freshwater ecosystems: a review. Environ. Chem. Lett. 20, 417–443. https://doi.org/10.1007/s10311-021-01341-5.
Reed, S., Clark, M., Thompson, R., Hughes, K.A., 2018. Microplastics in marine sediments near Rothera Research Station, Antarctica. Mar. Pollut. Bull. 133, 460–463. https://doi.org/10.1016/j.marpolbul.2018.05.068.
Rodrigues, M.O., Abrantes, N., Gonçalves, F.J.M., Nogueira, H., Marques, J.C., Gonçalves, A.M.M., 2019. Impacts of plastic products used in daily life on the environment and human health: what is known? Environ. Toxicol. Pharmacol. 72, 103239. https://doi.org/10.1016/j.etap.2019.103239.
Rodrigues, M.O., Gonçalves, A.M.M., Gonçalves, F.J.M., Abrantes, N., 2020. Improving cost-efficiency for MPs density separation by zinc chloride reuse. MethodsX 7, 100785. https://doi.org/10.1016/j.mex.2020.100785.
Santana, M.F.M., Kroon, F.J., van Herwerden, L., Vamvounis, G., Motti, C.A., 2022. An assessment workflow to recover microplastics from complex biological matrices. Mar. Pollut. Bull. 179, 113676. https://doi.org/10.1016/j.marpolbul.2022.113676.
Scheurer, M., Bigalke, M., 2018. Microplastics in Swiss floodplain soils. Environ. Sci. Technol. 52, 3591–3598. https://doi.org/10.1021/acs.est.7b06003.
Schrank, I., Möller, J.N., Imhof, H.K., Hauenstein, O., Zielke, F., Agarwal, S., Löder, M.G. J., Greiner, A., Laforsch, C., 2022. Microplastic sample purification methods - assessing detrimental effects of purification procedures on specific plastic types. Sci. Total Environ. 833, 154824. https://doi.org/10.1016/j.scitotenv.2022.154824.
Schröder, K., Kossel, E., Lenz, M., 2021. Microplastic abundance in beach sediments of the Kiel Fjord, Western Baltic Sea. Environ. Sci. Pollut. Res. 28, 26515–26528. https://doi.org/10.1007/s11356-020-12220-x.
Schütze, B., Thomas, D., Kraft, M., Brunotte, J., Kreuzig, R., 2022. Comparison of different salt solutions for density separation of conventional and biodegradable microplastic from solid sample matrices. Environ. Sci. Pollut. Res. 29, 81452–81467. https://doi.org/10.1007/s11356-022-21474-6.
Scott, J., Green, L., 2020. Development and Demonstration of a Superior Method for Microplastics Analysis: Improved Size Detection Limits, Greater Density Limits, and more Informative Reporting. TR Series (Illinois Sustainable Technology Center), pp. TR–078.
Skipp, G., L., Brownfield, I., 1993. Improved Density Gradient Separation Techniques Using Sodium Polytungstate and a Comparison to the Use of Other Heavy Liquids, Open-File Report.
Sruthy, S., Ramasamy, E.V., 2017. Microplastic pollution in Vembanad Lake, Kerala, India: the first report of microplastics in lake and estuarine sediments in India. Environ. Pollut. 222, 315–322. https://doi.org/10.1016/j.envpol.2016.12.038.
Stock, F., Kochleus, C., Bänsch-Baltruschat, B., Brennholt, N., Reifferscheid, G., 2019. Sampling techniques and preparation methods for microplastic analyses in the aquatic environment – a review. TrAC Trends Anal. Chem. 113, 84–92. https://doi.org/10.1016/j.trac.2019.01.014.
Stolte, A., Forster, S., Gerdts, G., Schubert, H., 2015. Microplastic concentrations in beach sediments along the German Baltic coast. Mar. Pollut. Bull. 99, 216–229. https://doi.org/10.1016/j.marpolbul.2015.07.022.
Thomas, D., Schütze, B., Heinze, W.M., Steinmetz, Z., 2020. Sample preparation techniques for the analysis of microplastics in soil—a review. Sustainability 12, 9074. https://doi.org/10.3390/su12219074.
Tirkey, A., Upadhyay, L.S.B., 2021. Microplastics: an overview on separation, identification and characterization of microplastics. Mar. Pollut. Bull. 170, 112604. https://doi.org/10.1016/j.marpolbul.2021.112604.
Torresan, M.E., 1987. The use of sodium polytungstate in heavy mineral separations report no. In: 87–590. Report, Open-File. https://doi.org/10.3133/ofr87590.
Tsering, T., Viitala, M., Hyvönen, M., Reinikainen, S.-P., Mänttäri, M., 2022. The assessment of particle selection and blank correction to enhance the analysis of microplastics with Raman microspectroscopy. Sci. Total Environ. 842, 156804. https://doi.org/10.1016/j.scitotenv.2022.156804.
Turner, S., Horton, A.A., Rose, N.L., Hall, C., 2019. A temporal sediment record of microplastics in an urban lake, London, UK. J. Paleolimnol. 61, 449–462. https://doi.org/10.1007/s10933-019-00071-7.
Van Cauwenberghe, L., Devriese, L., Galgani, F., Robbens, J., Janssen, C.R., 2015. Microplastics in sediments: a review of techniques, occurrence and effects. Marine Environmental Research, Particles in the Oceans: Implication for a safe marine environment 111, 5–17. https://doi.org/10.1016/j.marenvres.2015.06.007.
Vianello, A., Boldrin, A., Guerriero, P., Moschino, V., Rella, R., Sturaro, A., Da Ros, L., 2013. Microplastic particles in sediments of lagoon of Venice, Italy: first observations on occurrence, spatial patterns and identification. Estuarine, Coastal and Shelf Science, Pressures, Stresses, Shocks and Trends in Estuarine Ecosystems 130, 54–61. https://doi.org/10.1016/j.ecss.2013.03.022.
von der Esch, E., Lanzinger, M., Kohles, A.J., Schwaferts, C., Weisser, J., Hofmann, T., Glas, K., Elsner, M., Ivleva, N.P., 2020. Simple generation of Suspensible secondary microplastic reference particles via ultrasound treatment. Front. Chem. 8.
Waldschläger, K., Brückner, M.Z.M., Carney Almroth, B., Hackney, C.R., Adyel, T.M., Alimi, O.S., Belontz, S.L., Cowger, W., Doyle, D., Gray, A., Kane, I., Kooi, M., Kramer, M., Lechthaler, S., Michie, L., Nordam, T., Pohl, F., Russell, C., Thit, A., Umar, W., Valero, D., Varrani, A., Warrier, A.K., Woodall, L.C., Wu, N., 2022. Learning from natural sediments to tackle microplastics challenges: a multidisciplinary perspective. Earth Sci. Rev. 228, 104021. https://doi.org/10.1016/j.earscirev.2022.104021.
Waldschläger, K., Schüttrumpf, H., 2019. Effects of particle properties on the settling and rise velocities of microplastics in freshwater under laboratory conditions. Environ. Sci. Technol. 53, 1958–1966. https://doi.org/10.1021/acs.est.8b06794.
Wang, X., Bolan, N., Tsang, D.C.W., Sarkar, B., Bradney, L., Li, Y., 2021a. A review of microplastics aggregation in aquatic environment: influence factors, analytical methods, and environmental implications. J. Hazard. Mater. 402, 123496. https://doi.org/10.1016/j.jhazmat.2020.123496.
Wang, Z., Dou, M., Ren, P., Sun, B., Jia, R., Zhou, Y., 2021b. Settling velocity of irregularly shaped microplastics under steady and dynamic flow conditions. Environ. Sci. Pollut. Res. 28, 62116–62132. https://doi.org/10.1007/s11356-021-14654-3.
Wang, Z., Taylor, S.E., Sharma, P., Flury, M., 2018. Poor extraction efficiencies of polystyrene nano- and microplastics from biosolids and soil. PLoS One 13, e0208009. https://doi.org/10.1371/journal.pone.0208009.
Weber, F., Kerpen, J., 2023. Underestimating microplastics? Quantification of the recovery rate of microplastic particles including sampling, sample preparation, subsampling, and detection using μ-Ramanspectroscopy. Anal. Bioanal. Chem. 415, 2963–2973. https://doi.org/10.1007/s00216-022-04447-z.
Willis, K.A., Eriksen, R., Wilcox, C., Hardesty, B.D., 2017. Microplastic distribution at different sediment depths in an urban estuary. Frontiers in marine. Science 4.
Wright, S.L., Thompson, R.C., Galloway, T.S., 2013. The physical impacts of microplastics on marine organisms: a review. Environ. Pollut. 178, 483–492. https://doi.org/10.1016/j.envpol.2013.02.031.
Wu, M., Yang, C., Du, C., Liu, H., 2020. Microplastics in waters and soils: occurrence, analytical methods and ecotoxicological effects. Ecotoxicol. Environ. Saf. 202, 110910. https://doi.org/10.1016/j.ecoenv.2020.110910.
Yang, L., Zhang, Y., Kang, S., Wang, Z., Wu, C., 2021. Microplastics in freshwater sediment: a review on methods, occurrence, and sources. Sci. Total Environ. 754, 141948. https://doi.org/10.1016/j.scitotenv.2020.141948.
Yao, P., Zhou, B., Lu, Y., Yin, Y., Zong, Y., Chen, M.-T., O'Donnell, Z., 2019. A review of microplastics in sediments: spatial and temporal occurrences, biological effects, and analytic methods. Quaternary international, the 3rd ASQUA conference (part II) 519, 274–281. doi:https://doi.org/10.1016/j.quaint.2019.03.028.
Yu, Y., Kumar, M., Bolan, S., Padhye, L.P., Bolan, N., Li, S., Wang, L., Hou, D., Li, Y., 2024. Various additive release from microplastics and their toxicity in aquatic environments. Environ. Pollut. 343, 123219. https://doi.org/10.1016/j.envpol.2023.123219.
Zhang, S., Yang, X., Gertsen, H., Peters, P., Salánki, T., Geissen, V., 2018. A simple method for the extraction and identification of light density microplastics from soil.

Sci. Total Environ. 616–617, 1056–1065. https://doi.org/10.1016/j.scitotenv.2017.10.213.
Zhang, X., Yu, K., Zhang, H., Liu, Y., He, J., Liu, X., Jiang, J., 2020. A novel heating-assisted density separation method for extracting microplastics from sediments. Chemosphere 256, 127039. https://doi.org/10.1016/j.chemosphere.2020.127039.
Zhang, Y., Jiang, H., Bian, K., Wang, H., Wang, C., 2021. A critical review of control and removal strategies for microplastics from aquatic environments. J. Environ. Chem. Eng. 9, 105463. https://doi.org/10.1016/j.jece.2021.105463.
Zheng, Y., Li, J., Cao, W., Liu, X., Jiang, F., Ding, J., Yin, X., Sun, C., 2019. Distribution characteristics of microplastics in the seawater and sediment: a case study in Jiaozhou Bay, China. Sci. Total Environ. 674, 27–35. https://doi.org/10.1016/j.scitotenv.2019.04.008.
Zobkov, M., Esiukova, E., 2017. Microplastics in Baltic bottom sediments: quantification procedures and first results. Mar. Pollut. Bull. 114, 724–732. https://doi.org/10.1016/j.marpolbul.2016.10.060.